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# Polymer injection molding of hard X-ray refractive optics

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Refractive lenses are versatile optical components in synchrotron radiation beamlines and act e.g. as condensers or objectives in hard ( $E > 10$  keV) X-ray microscopes [1]. X-ray lenses are characterized by having a low refractive power and a high absorption. Their radii of curvature (ROC) are typically in the lower micron range, many single elements are put in series and they are made out of low-Z materials (Fig. 1). The demand on their shapes is high and hence microlithography combined with deep reactive ion etching (DRIE) of silicon is a favorable fabrication route. Although silicon is an uncontested lens material, the optical performance of a silicon X-ray lens is ultimately limited by absorption.

Here, we present a new route for X-ray lens manufacture: polymer injection molding. The optical advantages of using a thermoplastic polymer are striking; at photon energies around 17 keV and a focal length of 200 mm the achievable spot size may be halved, the effective aperture may be tripled and the gain in photon flux may be increased tenfold considering line focusing compared to the silicon equivalent.

Polymeric X-ray lenses were developed at DTU Danchip, the microfabrication facility of the Technical University of Denmark. A silicon master was manufactured, from which a nickel electroform was obtained, which in turn was used as an insert in an injection-molding tool.

The manufacture of the silicon master included standard UV-lithography and pattern transfer by reactive ion etching into a thermally grown silicon oxide layer, which served as a hard mask for subsequent deep reactive ion etching of the silicon using an optimized Bosch process. The features are basically cavities in the form of parabolic cylinders with radii of curvature of nominally 50  $\mu\text{m}$ , lateral dimensions of 450  $\mu\text{m} \times 300 \mu\text{m}$  and, depths of 200  $\mu\text{m}$  (Fig.2). The most challenging process is DRIE, where the main target is to achieve close to 90° sidewalls along the whole feature in order to guarantee lens uniformity and successful release of the polymeric part from the mold. The three-dimensional shapes of the lens cavities were characterized by optical profilometry and by a previously reported procedure based on replica molding and atomic force microscopy [2].

To obtain the nickel mold insert, the silicon master was first uniformly covered with a thin layer of amorphous silicon by low pressure chemical vapor deposition, next sputter coated with a metal seed layer and then conformably electroplated with nickel. The silicon wafer was dissolved in aqueous potassium hydroxide and the 600  $\mu\text{m}$  thick nickel mold insert containing the inverse lens structures was cut by laser micro machining (Fig.3 & Fig.4).

Polymer chips comprising multiple arrays of X-ray lenses were injection molded in an ENGEL Victory Tech injection molding machine (Fig.5 & Fig.6). A variotherm process with a cycle time of 3 min was optimized. Polymers like polypropylene or polyethylene that are more flexible and have high mold shrinkage upon solidification, showed the highest chances of being successfully released from the nickel shim. The most critical region was the web between the lens cavities, where the local aspect ratio approaches 10. Mechanical stress on the polymer part when it was pulled away from the nickel shim sometimes deformed the webs or even caused their rupture.

The X-ray optical performance has been tested at the European Synchrotron Radiation Facility, ID06. A preliminary test of a thermoplastic lens showed a 60  $\mu\text{m}$  long line beam with a waist of 700 nm, a gain of 50 and a total transmission of 30% at 17 keV. Polymer injection molding in combination with UV lithography and DRIE has proven to be a viable route for obtaining high efficient X-ray optics at low cost.

- [1] Simons, H. King, A. Ludwig, W. Detlefs, C. Pantleon, W. Schmidt, S. Stöhr, F. Snigireva, I. Snigirev, A. Poulsen, H.F. Nat. Commun. 6, 6098 (2015).
- [2] Stöhr, F. Michael-Lindhard, J. Simons, H. Poulsen, H.F. Hübner, J. Hansen, O. Garnaes, J. Jensen, F. Microelectron. Eng. 141, 6-11 (2015).

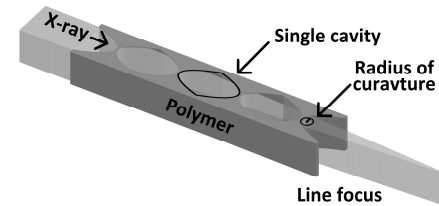


Fig. 1. Concept of a compound lens focusing X-rays into a line. Individual elements have a concave shape, since the refractive index for hard X-rays is smaller than 1. Multiple lenses are put in series to increase the refractive power.

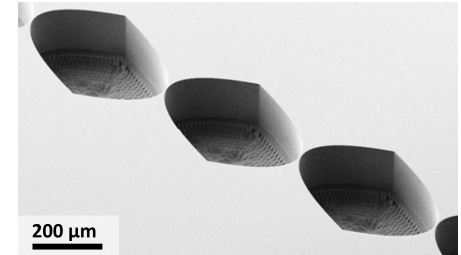


Fig. 2. SEM of the silicon master structures after DRIE, removal of the sacrificial structures and surface smoothing.

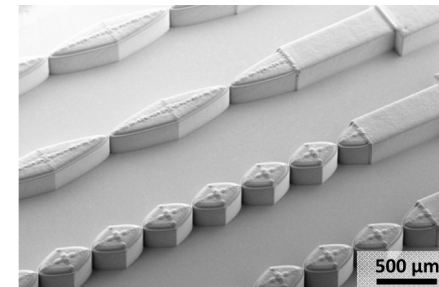


Fig. 3. SEM of a section of the nickel mold insert comprising arrays of bi-parabolic structures with radii of curvature of 20  $\mu\text{m}$  and 50  $\mu\text{m}$ .

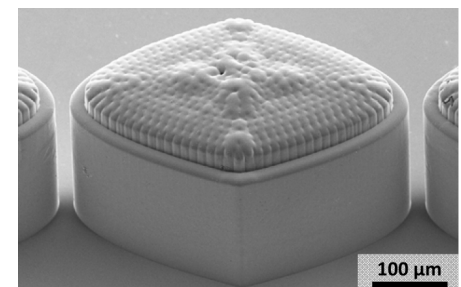


Fig. 4. SEM of a single nickel feature. Note the straightness and smoothness of the sidewalls. Sacrificial structure residuals are seen on the top.

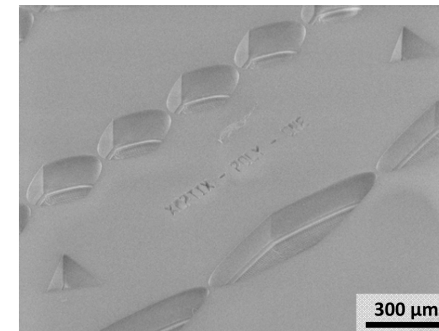


Fig. 5. SEM of a section of a polymer lens chip comprising arrays of lens elements with a radius of curvature of 20  $\mu\text{m}$  and 50  $\mu\text{m}$ .

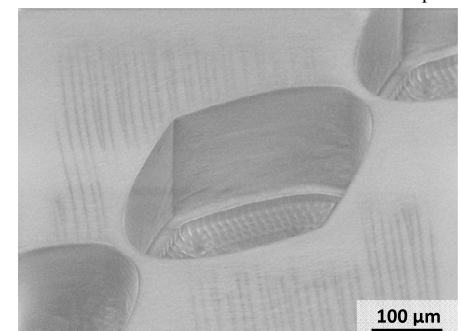


Fig. 6. SEM of a polymer cavity.